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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF:

SUGIO Youko, et al.

SERIAL NO.: 10/667,304

FILED: SEPTEMBER 23, 2003

FOR: PRODUCTION PROCESS OF 2,7-DIBROMOFLUORENONE

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: EXAMINER: WITHERSPOON, S. A.

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: GROUP ART UNIT: 1621

DECLARATION UNDER 37 C.F. R. §1.132

COMMISSIONER FOR PATENTS  
ALEXANDRIA, VA 22313-1450

SIR:

Now comes SUGIO Youko, who deposes and states that:

1. That I am an inventor of the above-identified application.

2. That I am a graduate of Yamaguchi University at Yamaguchi Prefecture, Japan, and received my Bachelorship in the field of the industrial chemistry, in the year 1998.

3. That I have been employed by The ADCHEMCO CORPORATION for 5 years and The JFE CHEMICAL CORPORATION ( the former corporation is the same entity as the one of the latter ) for one year as a researcher in the field of the industrial chemistry.

4. That I understand the English language or, at least, that the contents of the Declaration were made clear to me prior to executing the same.

5. That the following experiment was carried out by me.

Experiment

6. The following experiments demonstrate the comparison of the production

process of 2,7-dibromofluorenone in the presence of a caustic compound according to this invention with the comparative one in the absence of the caustic compound.

7-1. The reaction in the presence of sodium hydroxide (the same reaction as that of example 1 in the specification of the present invention.).

Into a three-necked 300-ml-flask equipped with a stirrer, a molecular-oxygen-containing gas (air) inlet tube and a gas outlet tube (fitted with a condenser), 2,7-dibromofluorene(9.29g, 0.029 mol) was charged, followed by its dissolution with toluene (100 g). Subsequent to addition of tetraammonium bromide (0.24 g) and a 48% aqueous solution of sodium hydroxide (2.26 g), the resulting mixture was vigorously stirred at 60°C for 2 hours while introducing air at a flow rate of 0.1 L/min. After completion of the stirring, the reaction mixture was allowed to stand, and the resulting toluene layer was collected. The toluene layer was then subjected to acid washing, followed by water washing until its pH arose to 7. Toluene was then distilled off to recover crude crystals of 2,7-dibromofluorenone. The content of 2,7-dibromofluorene in the crude crystals at that time was 0%. Those crude crystals were recrystallized from N,N'-dimethylformamide (15 g) to afford 2,7-dibromofluorenone as yellow crystals (8.20 g). Purity: 99.4%, yield: 89.0%, melting point: 206°C.

7-2. The comparative reaction in the absence of sodium hydroxide

The reaction was conducted in the same way and experimental conditions as the above ones except for no addition of an aqueous solution of sodium hydroxide. The crystals of 2,7-dibromofluorenone were, however, scarcely obtained; further 2,7-dibromofluorenone was incapable of being detected in the reaction mixture by a gas chromatographic method with a column of DB-5; even in the mixture with a reaction time prolonged 4 hours longer than that of the above reaction; these results were unexpected for the petitioner.

8. Based on the above results, the caustic alkali compound is concluded to be an

essential component for the formation of 2,7-dibromofluorenone from 2,7-dibromofluorene in the present invention.

9. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

10. Further deponent saith not.

Sugio Youko  
Signature      SUGIO YUUKO

September 16, 2004  
Date      September 16, 2004